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Light-free magnetic resonance force microscopy for studies of electron spin polarized systems

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Abstract

Magnetic resonance force microscopy is a scanned probe technique capable of three-dimensional magnetic resonance imaging. Its excellent sensitivity opens the possibility for magnetic resonance studies of spin accumulation resulting from the injection of spin polarized currents into a para-magnetic collector. The method is based on mechanical detection of magnetic resonance which requires low noise detection of cantilever displacement; so far, this has been accomplished using optical interferometry. This is undesirable for experiments on doped silicon, where the presence of light is known to enhance spin relaxation rates. We report a non-optical displacement detection scheme based on sensitive microwave capacitive readout.

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1. Introduction

The magnetic resonance force microscope (MRFM) is a novel scanned probe instrument that detects magnetic resonance signals from well-defined *subsurface* volumes in a wide variety of materials. Unlike conventional magnetic reso-

nance techniques that inductively detect the processing spin moments, MRFM detects magnetic resonance through the force exerted by the spin magnetization on a micromechanical cantilever. The key element of an MRFM is the micromechanical resonator, typically a microcantilever, with a micromagnetic probe mounted on its end (see Fig. 1). This probe magnet generates a nonuniform magnetic field and, through the gradient of that field, magnetically couples to

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Fig. 1. Geometry of the MRFM experiment. A sample is placed directly on a microstrip resonator used to manipulate the electron spin magnetization. A force sensing cantilever with a micromagnetic probe tip is brought in close proximity to the sample. The cantilever is, in turn, capacitively coupled to a microwave sensing resonator used for capacitive displacement detection. The entire assembly is placed in an external magnetic field H_{ext} .

magnetic moments (spins) in the sample under study. The probe magnet also serves one more important purpose: it enables subsurface imaging with MRFM. As in conventional magnetic resonance imaging, the nonuniform magnetic field selects the spatial region (or "sensitive slice"), under the surface of a sample, where the magnetic resonance condition is satisfied. Only spins located in this slice will be manipulated by means of standard microwave/RF manipulation techniques. Thus, the MRFM will interact with an ensemble of spins residing in a well-defined spatial location. This subsurface imaging capability sets MRFM apart from other existing scanning probe techniques which provide information only about the sample surface.

Another advantage of the MRFM is its unprecedented sensitivity. The MRFM signal is detected by measuring the force of interaction between the probe magnet and spins in the sample; sufficiently sensitive measurement of the resulting displacement of the cantilever ensures that the ultimate sensitivity of the method is limited only [1] by the thermal noise of the mechanical resonator. It has been estimated that detection of the signal from a single electron spin is possible, however the required sensitivity is at least 9–10 orders of magnitude better than that of conventional EPR equipment.

A typical force of interaction of a probe magnet with a single electron spin, achievable with a realistic magnetic field gradient, is of order 1 aN. It can be easily seen that the resulting displacement produced by such a force applied harmonically at the resonant frequency of the cantilever is 10^{-12} – 10^{-14} m; this sets the cantilever displacement readout sensitivity requirements for a single electron spin MRFM experiment.

In all reported MRFM experiments, cantilever displacement has been detected by means of a fiber optic interferometer. Such a device is very easy to operate, is compatible with vacuum and cryogenic environments and routinely provides displacement sensitivity of 10^{-13} m/ $\sqrt{\text{Hz}}$ which is adequate for a single electron spin experiment.

The extremely high sensitivity of MRFM with the ability to detect a range of magnetic resonance phenomena (EPR, NMR, FMR) makes it an invaluable tool for the study of many types of physical systems. An important class of such systems requires the detection of electron spin resonance in semiconductors. An example of such a system is phosphorus-doped silicon (Si:P). Such a capability is needed for dopant profile mapping in semiconducting devices or for characterization and operation of a solid-state quantum computer [2]. However, the relaxation time of electron spins on phosphorus donors in Si is strongly affected by the presence of light [3]. Irradiation of a sample with even small intensities of sub-band gap light, which is still capable of ionizing shallow phosphorus donors, can reduce the electron relaxation time by several orders of magnitude. A conventional MRFM setup based on optical cantilever displacement detection unavoidably introduces unwanted stray light. Therefore, there is a call for a detection scheme that does not employ light.

In order to satisfy this need, we have implemented a displacement detector based on capacitive detection. The detector measures the change in the capacitance between the cantilever and a sensing electrode caused by the cantilever displacement.

2. Principles of operation

The arrangement is based on a capacitance detection technique presented in Ref. [4]. Fig. 2 shows a schematic: a resonant microwave circuit is capacitively coupled via a sensing wire to a micromechanical cantilever such that displacement of the cantilever results in a change of the coupling capacitance and thus a change of the resonant frequency of the resonator. This frequency change is monitored using standard microwave phaseshift detection techniques.

As the resonant circuit is capacitively coupled to a grounded cantilever, its resonant frequency will be

$$\omega_0 = \sqrt{\frac{1}{L[C_{\rm res} + C_p(z)]}},$$

where L is the inductance of the resonant tank circuit, C_{res} is its capacitance and $C_p(z)$ is the capacitance between the sensing wire and the cantilever; the latter depends on their separation z.

For small displacements of the cantilever δz about its equilibrium position z_0 , $C_p(z)$ can be approximated as

$$C_p(z) = C_p(z_0) + \frac{\mathrm{d}C_p}{\mathrm{d}z}\Big|_{z=z_0} \delta z.$$

Using this result, the resonant frequency shift of the loaded tank circuit due to small cantilever



Fig. 2. Schematic diagram of the capacitive displacement detection circuit. A microwave resonant structure, presented here as a lumped LC tank circuit, is coupled to a micro-mechanical cantilever via a sensing wire. The tank circuit is driven at its loaded resonant frequency ω_0 with a 50 Ω harmonic voltage source of amplitude V_{osc} . A standard microwave mixer-based detection technique is used to detect frequency shifts associated with cantilever motion.

displacements is

$$\delta\omega = \omega_0 \left. \frac{1}{2[C_{\text{res}} + C_p(z_0)]} \frac{\mathrm{d}C_p}{\mathrm{d}z} \right|_{z=z_0} \delta z. \tag{1}$$

The ultimate sensitivity with which this change can be measured is limited only by the thermal charge fluctuations in the tank circuit. Thus, if the microwave resonant circuit is driven at its loaded resonant frequency ω_0 with a harmonic voltage of amplitude V_{osc} , the minimum detectable frequency change $\delta\omega$ is given by

$$\delta\omega = \sqrt{\frac{\omega_0 k_{\rm B} T \Delta v}{Q[C_{\rm res} + C_p(z)] \langle V_{\rm osc}^2 \rangle}},\tag{2}$$

where $k_{\rm B}$ is Boltzmann's constant, T is the temperature of the microwave resonator, Δv is the bandwidth measurement, Q is the quality factor of the microwave resonator and $\langle V_{\rm osc}^2 \rangle$ is the average squared amplitude of the drive voltage.

With the help of a parallel-plate capacitor model the probe capacitance and its first derivative can be estimated as

$$C_p = \varepsilon_0 \, \frac{A}{z},\tag{3}$$

and

$$\frac{\mathrm{d}C_p}{\mathrm{d}z}(z) = -\varepsilon_0 \,\frac{A}{z^2},\tag{4}$$

where A is the effective area of the capacitor plates.

Thus, the minimum detectable displacement of the cantilever can be obtained by combining Eqs. (1)-(4):

$$\delta z_{\min} = \frac{2z_0^2}{\varepsilon_0 A} \sqrt{\frac{\left(C_{\rm res} + \varepsilon_0 \frac{A}{z_0}\right) k_{\rm B} T \Delta \nu}{\omega_0 Q \langle V_{\rm osc}^2 \rangle}}.$$
 (5)

Eq. (5) shows the experimental parameters that can be tuned to improve the sensitivity of the method. In practice, the amplitude of the driving voltage can be increased and the gap z_0 of the probe capacitor can be minimized. Other parameters cannot be changed as easily. For example, the area A is defined by the dimensions of a cantilever used in the experiment and the temperature of operation is defined by the conditions of the MRFM experiment for which the capacitive displacement detection is used.

Using typical parameters, the sensitivity of the method can be estimated to be as good as 10^{-13} – 10^{-14} m/ $\sqrt{\text{Hz}}$ at T = 300 K, comparable to the sensitivity obtained with optical interferometry.

3. Experimental setup

Our detector is based on a 2.5 GHz microwave resonator, this choice of frequency represents a compromise between detector performance, ease of operation and equipment costs. Making a tank circuit with such a high resonant frequency out of lumped elements, as shown in Fig. 2, is difficult. Instead, we implemented it as a $\lambda/4$ microstrip resonator based on an existing design presented in Ref. [5]. The arrangement is shown schematically Fig. 1. An AFM cantilever, coated with a 10 nm Au layer to facilitate its grounding is coupled to the resonator via a short (less than 1 mm) 25 µm diameter gold wire. This coupling does not significantly affect the resonant properties of the microwave resonator. The new resonant frequency of the loaded resonator is typically reduced to $2.45 \pm 0.02 \,\text{GHz}.$

The output of a microwave source [6] tuned to the resonant frequency of the loaded resonator is split, with half used as a local oscillator signal for a microwave mixer and the other half used to drive the loaded microwave sensing resonator. The signal transmitted through the resonator is applied to the RF input of the mixer. The magnitude of the resulting low-frequency signal at the IF output of the mixer is proportional to the phase difference between the transmitted wave and the local oscillator. Thus, cantilever motion shifts the resonant frequency of the microwave resonator, which in turn shifts the mixer output signal.

For testing, a cantilever was mounted on a piezo that was excited with a harmonic voltage of known amplitude V_{osc} and frequency. The resultant cantilever motion modulates the mixer output; this signal is detected with a lock-in amplifier [7] using the excitation signal as a reference. The frequency response of the cantilever oscillation is

traced by sweeping the excitation signal through the mechanical resonance of the cantilever. Calibration of the cantilever response is based on the known displacement amplitude of the cantilever A_{res} at its resonant frequency, where

$$A_{\rm res} = d_{33} V_{\rm osc} Q,\tag{6}$$

where $d_{33} = 4.8 \text{ Å/V}$ is the coefficient of the EBL#6 piezoelectric material [8] used to excite the motion and Q is the measured cantilever quality factor.

Using this approach, detection of the displacement of various types of MRFM cantilevers has been demonstrated at room temperature. As an example, Fig. 3 shows the frequency response of a triangular cantilever custom fabricated for MRFM experiments at the Laboratory for Physical Sciences. This cantilever was driven with a piezo excitation amplitude of 4.8×10^{-3} Å. We find a readout sensitivity of approximately $5.0 \times 10^{-12} \text{ m/}\sqrt{\text{Hz}}$; in this case the overall detector noise level is dominated by displacement readout noise which corresponds to a force sensitivity of $80 \text{ aN}/\sqrt{\text{Hz}}$.

In order to verify the applicability of this displacement detection method for MRFM experi-



Fig. 3. Capacitively detected response of a triangular cantilever fabricated at the Laboratory for Physical Sciences to a piezo excitation amplitude of 4.8×10^{-3} Å. We find a readout sensitivity of approximately $5.0 \times 10^{-12} \text{ m}/\sqrt{\text{Hz}}$; in this case the overall detector noise level is dominated by displacement readout noise which corresponds to a force sensitivity of $80 \text{ aN}/\sqrt{\text{Hz}}$. The signal was acquired at T = 300 K in vacuum.



Fig. 4. ESR–MRFM signal detected via capacitive displacement detection. The signal is detected from a 2,2-diphenyl-1picrylhydrazyl free radical (DPPH) sample at 4 K. This signal is detected with ESR microwave excitation frequency of 7.858 GHz. The insert shows the linear field dependence of the ESR resonant frequency with a corresponding g factor of 2.012.

ments, the system was installed in a ³He cryostat, where it was successfully tested at low temperatures in vacuum. Fig. 4 demonstrates the first lightfree detection of an MRFM signal; in this case the ESR signal from 2,2-diphenyl-1-picrylhydrazyl (DPPH) detected at T = 4 K with ESR magnetic resonance frequency of 7.858 GHz. The variation of the magnetic resonance frequency varied in proportion to the applied magnetic field as shown in the inset to Fig. 4, with a proportionality constant corresponding to g = 2.012 consistent with the known value for DPPH.

4. Discussion and future work

The power of capacitive displacement readout for application in a low-temperature MRFM is evident. Its efficacy in a laboratory microscope will be improved by increasing its sensitivity and ease of use; particularly important is improving applicability at low temperature.

The sensitivity of the method is strongly dependent on the capacitive gap z_0 between the sensing electrode and the cantilever. Whereas the

size of this gap and the position of the cantilever relative to the sensing wire can be easily controlled at room temperature, as the system is cooled down to cryogenic temperatures, this alignment can be significantly disturbed by the thermal drift of the cantilever and/or the sensing wire, thus degrading sensitivity. This challenge can be much improved through attention to this issue in the design of cantilever mounting system.

Heating due to the microwave power applied to the sensing resonator can be minimized by several means at cryogenic temperatures. At 4 K where the resonator was operated at $\approx 5.0 \, \text{dBm}$, we did not observe any significant temperature increase in the system, but the situation might change if the experiment is to be conducted at sub-kelvin temperatures where the cooling power of a ³He refrigerator is limited to a few mW. We will reduce heating by fabricating the microwave sensing resonator out of superconducting material and designing the resonator to minimize electromagnetic radiation. A design that shields the displacement sensing resonator will also protect it from the microwave resonator used in MRFM experiment for spin manipulation Fig. 1 to minimize unwanted cross-talk between these.

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References

- [1] J.A. Sidles, D. Rugar, Phys. Rev. Lett. 70 (1993) 3506.
- [2] B.E. Kane, Nature 393 (1998) 133.
- [3] G. Feher, E.A. Gere, Phys. Rev. 114 (1959) 1245.
- [4] R.C. Palmer, E.J. Denlinger, H. Kawamoto, RCA. Rev. 43 (1982) 195.
- [5] T. Tran, D.R. Oliver, D.J. Thomson, G.E. Bridges, Rev. Sci. Instrum. 72 (2001) 2618.
- [6] Agilent model 8648C.
- [7] Perkin Elmer Instruments 7280 DSP lock-in amplifier.
- [8] Staveley NDT Technologies, http://www.staveleyndt.com/ products/ebllzt.html.